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मानक

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Mazdoor Kisan Shakti Sangathan

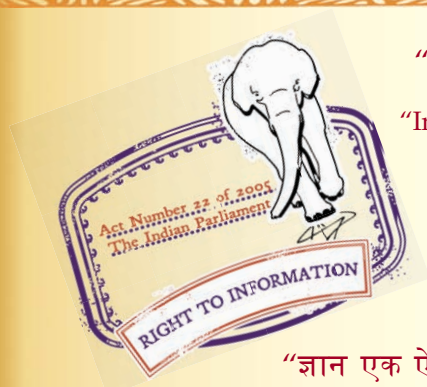
“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 12686 (1989): Abrasives - Diamond or Cubic Boron Nitride
Micron Powder Sizes - Testing [PGD 9: Abrasives]



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“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**ABRASIVES—DIAMOND OR CUBIC BORON
NITRIDE MICRON POWDER SIZES**

भारतीय मानक

अब्रेजिब-होरे या घनाकार बोरान नाइट्राइड माइक्रॉन चूर्ण साइज

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 12 April 1989, after the draft finalized by the Abrasives Sectional Committee had been approved by the Mechanical Engineering Division Council.

Grain sizes of diamond powders are distinguished in macro and micro grain sizes. In this standard, micro grain sizes have been covered. For macro-grain sizes, reference may be made to IS 10960 (Part 1) : 1984 'Grain sizes for diamond or cubic boron nitride: Part 1 Designation and grain size distribution', and IS 10960 (Part 2) : 1984, Part 2 'Methods of test of macrogrits'.

This standard provides a recognized standard method for the sizing of diamond/cubic boron nitride powder in micron (μm) sizes. It also serves a common basis for understanding between the purchaser and the seller as to the quality and particle size desired or supplied.

While preparing this standard, assistance has been taken from the following:

ANSI B 74-20-1981 Grading of diamond powder in sub-sieve sizes, American National Standard Institute, USA, and

FEPA — Nov. 1977 Standard for diamond micron powder sizes. Federation Europeenne des Fabricants de Produits Abrasifs (FEPA).

Indian Standard

ABRASIVES—DIAMOND OR CUBIC BORON NITRIDE MICRON POWDER SIZES

1 SCOPE

This standard covers the requirements of powder sizes of diamond (natural or synthetic) or cubic boron nitride micron powders, standard size ranges of these powders and also the method of measuring the particle size based on the use of an optical microscope.

NOTE — For simplification, reference is made throughout this standard to 'Diamond' grains but all indications also apply to cubic boron nitride.

2 REFERENCES

- IS 10960 Grain sizes for diamonds or cubic boron nitride:
 (Part 1) : 1984 Part 1 Designation and grain size distribution.
 (Part 2) : 1984 Methods of test of macro grits

3 TERMINOLOGY

3.1 Fines (Undersize Particles)

Fines (*undersize particles*) are any particles smaller than the minimum for a specified size.

3.2 Micron Powder Sizes

These are size classified products of natural or synthetic diamond and constitute those sizes usually prepared by methods of separation other than sieving. These are usually smaller than the finest sieve size.

3.3 Particle Size

It is defined in terms of measurements made on the magnified image of the particle as seen in the microscope. The size is determined from the diameter of the smallest circle which completely circumscribes the image.

3.4 Shales

A shale is a particle of thin plate like shape, which when viewed through a microscope using transmitted light, appears to be transparent.

3.5 Slivers

A sliver is a particle of needle or rod-like shape such that the ratio of the major to the minor axis is greater than 3 : 1.

4 REQUIREMENTS

4.1 General

All diamond powders designated or represented as complying with this standard shall meet all the requirements given in this standard.

4.2 Standard Powder Sizes

The standard size ranges, size designations and size tolerances shall be as given in Table 1. Other sizes may also be made provided the powder complies with all other requirements of this standard and the exact size range and maximum oversize is clearly stated on the labels of the packings.

4.3 Material

The powder shall consist of crushed diamond, cleaned of all impurities, meeting the requirements given in 4.6.

4.4 Slivers

The micron sizes shall not contain any slivers that are longer than twice the maximum oversize for any specified size range but may contain up to 3 percent by count of slivers that have lengths up to twice the maximum oversize.

4.5 Shales

The micron sizes shall contain not more than 10 percent by count of shales, disregarding fines.

4.6 Impurities

A maximum of 2 percent by weight of impurities shall be permitted.

5 PROCEDURE

5.1 Sample

A representative sample of the material to be tested is prepared in a form suitable for the instrument to be used for inspection. If this is an optical microscope, the slide must be prepared by the method specified in 6.1.

5.2 Preparation for Inspection

The microscope must be properly aligned and the magnification determined before the inspection is commenced. Alignment procedures

Table 1 Standard Micron Powder Sizes
(Clause 4.2)

All dimensions in millimetres.			
Size Designation (1)	Permissible Range for the Mean Size μm^* (2)	Maximum Standard Deviation μm^* (3)	Maximum Particle Size μm^* (4)
M 1.0	0.9 - 1.1	0.28	2.8
M 1.6	1.4 - 1.8	0.28	4.5
M 2.5	2.2 - 2.8	0.28	6.5
M 4.0	3.6 - 4.4	0.26	10
M 6.3	5.6 - 7.0	0.24	15
M 10	9 - 11	0.28	24
M 16	14 - 18	0.22	35
M 25	22 - 28	0.22	55
M 40	36 - 44	0.20	80
M 63	56 - 70	0.20	126

1 micrometre = 1 μm = 1 micron = 10 m.

*For the method of calculations, refer Annex A.

will vary from instrument to instrument but must be such as to ensure optimum performance. The magnification is to be checked by the use of a stage micrometer of known accuracy. It must be determined within maximum error limits of ± 0.5 percent. Further requirements concerning the actual values of magnification to be used are given in Table 2. Reference may also be made to Annex B which lists requirements for the microscope system.

5.3 Inspection

In total, 400 particles are to be measured. The methods of doing it and of ensuring operator reliability are given in 6.2. The method makes use of a transparent graticule which carries a series of circles of accurately known diameters. The graticule is superposed on the image of the powder and the particle sizes are matched to the graticule circles as described in 6.2.

6 TEST METHOD

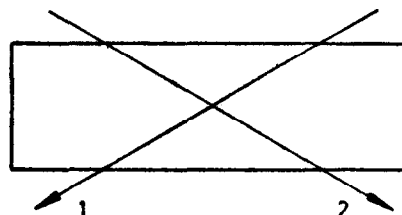
6.1 Sample Preparation

The powder shall be thoroughly mixed and samples taken with a clean micro-spatula, or other fine object, from four or five locations, the aliquots then being combined on a clean glass slide. Because of their very small size and their tendency to form electrostatic aggregates, the handling and sampling of diamond micron powders presents problems, particularly if potentially harmful contamination from larger particles is to be avoided. This standard does not specify handling procedures, but it is imperative that high standards of cleanliness be

maintained during all the stages described in this standard.

Suitable microscopic slides are prepared by mixing a small quantity of *Canada Balsam* of suitable consistency with the specimen so collected. The powder should be thoroughly blended with a freshly pointed stick which shall be moved in a spiral fashion away from and towards the centre of the slurry in order to prevent size differentiation. The diameter of the slurry so prepared may conveniently be 10 to 15 mm.

A representative sample is taken from the slurry on the tip of a freshly sharpened stick by moving the point of the stick first in direction 1 and then in direction 2 as shown below:



The material so removed is placed on a single drop of *Canada Balsam* on a separate slide and is blended in circular fashion into a circle of approximately 5 mm diameter.

A clean cover glass is placed over the slurry. The under side of the slide is gently heated to allow the particles to settle and the cover glass is compressed with gentle pressure from a clean

Table 2 Required Magnification Settings
(Clauses 5.2, B-3.2 and B-3.3)

Size Designation	Magnification	Approximate Main Range of Image Sizes mm	Oversize Limit at Recommended Magnification mm
M 1.0	2 500 ×	1 – 4	7
M 1.6	2 500 ×	2.5 – 6	11
M 2.5	2 500 ×	4 – 10	16
M 4.0	2 500 ×	5 – 16	25
M 6.3	1 600 ×	6 – 16	24
M 10	1 000 ×	6 – 15	24
M 16	625 ×	6 – 15	22
M 25	400 ×	6 – 15	22
M 40	250 ×	6 – 15	20
M 63	160 ×	6 – 15	20

soft object (for example, a pencil eraser) while the assembly is still warm, to provide a monolayer of particles. In this final slide, the *Balsam* must be wholly retained within the area of the cover glass. If this is not the case, a new slide must be prepared using smaller quantities of sample slurry and of *Canada Balsam*.

The slide will be most suitable for inspection if the particle concentration is such that about twenty particles per field are seen at the magnification employed for measurement. The particles shall be well separated and without aggregation. If there is significant aggregation, the slide must be rejected and a new one prepared.

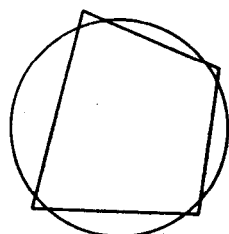
6.2 Measurement Procedure

6.2.1 Use of Graticule

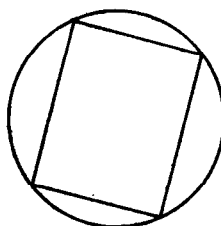
The screen graticule is placed over the magnified particle image until the appropriate circumscribing circle is determined as shown below:

- A: The circle is just too small.
- B: The circle is the smallest which circumscribes the particle. The inner edge of the circle is definitive.
- C: The circle is too large.

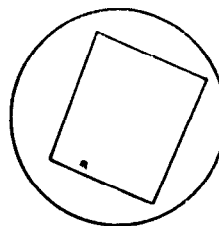
Thus the size is determined by circle B.



A



B



C

A proper assignment is made for each particle examined, up to the requisite number of particles, and a record kept of the frequency of occurrence of particles in every size interval. The relative numbers of particles in each interval when expressed as a percentage of the total number, constitute the size distribution by number.

All particles which are totally visible within any selected field shall be counted except those particles in contact with the extremities of the field of view which shall be defined by lines on the projection screen. These lines shall be not less than 15 mm from the outer limit of the visible image. A sufficient number of areas from different zones of the slide shall be measured up to the total required number of particles.

Despite all care in sample preparation, it is still possible that some segregation of size occurs between centre and periphery of the preparations. Therefore, the areas of the slide for measurement shall be chosen to include central and peripheral areas equally. A suggested method is to traverse the preparation from periphery to centre along a radius, choosing areas at equal intervals and repeating along other radii as necessary, to come to the desired total.

6.2.2 Operator Training and Selection

A well known feature of microscopic inspection is that different observers experience different visual sensation upon examining a selected image. In situations where a single magnified (projected) image is to be measured, a variety of measurements may be reported, for the same image, by individual observers. The reasons underlying the differences between observers concern the basic physiological differences between individuals, in the way they receive electrical signals at the rear of the eye, and in the way these signals are subsequently processed by the brain. It has been shown that individual observers measure images consistently but differently, and that people may be grouped into several classes depending upon how they observe images.

For the purpose of this standard, it is imperative that such inter-operator differences be reduced to the minimum and for this reason, the following operator training schedule is required:

- a) Select a standard training graticule. This is an artificial sample which has been reproduced by photographic means and simulates an actual diamond powder sample. Place this on the stage of the

microscope (after determining the appropriate microscope magnification factor) and focus the image.

- b) The operator shall classify magnified artificial particle image using the method of this standard.
- c) Calculate the two distribution parameters, mean size and standard deviation, from this count data.
- d) Compare the actual values of mean size and standard deviation determined for the artificial sample by the operator being tested with the values supplied with the graticule. These values are averages of the results of a large number of experienced operators.
- e) If the measured mean value differs from the value supplied by more than 2 percent, or if the standard deviation exceeds the indicated limit, the results are suspect and the operator recheck his/her procedure. Operators who consistently fail to obtain results within these limits shall not be used for checking powders for compliance with this standard.

ANNEX A

(Table 1)

CALCULATION OF DISTRIBUTION PARAMETERS FROM THE NUMBER SIZE DISTRIBUTION

A-1 DETERMINATION OF MEAN SIZE

A-1.1 Consider the hypothetical but typical number size distribution as shown in Table 3.

The mean size is calculated by summing up the product of class midpoints and the number of particles in that class, for the total number of occupied classes, and dividing by the total number of particles counted.

Thus, $\bar{X}$ the mean size is given by

$$\frac{1}{N} \sum (x_i f_i)$$

where

N = total number of particles observed,

x_i = midpoint of i th class interval, and

f_i = frequency of i th class interval.

Thus in this case, where $N = 400$, the mean size is given by:

$$\bar{X} = \frac{1}{400} \left\{ \begin{aligned} &[(6.5 \times 11) + (7.5 \times 29) + \\ &(8.5 \times 68) + (9.5 \times 92) + \\ &(10.5 \times 96) + (11.5 \times 64) + \\ &[(12.5 \times 30) + (13.5 \times 10)] \end{aligned} \right\}$$

$$= 9.99 \mu\text{m.}$$

A-2 DETERMINATION OF STANDARD DEVIATION

A-2.1 The standard deviation, S , of any distribution is given by the expression:

$$S = \left[\frac{\sum f_i (x_i - \bar{x})^2}{N} \right]^{\frac{1}{2}}$$

which for ease of commutation reduces to:

$$S = \left[\frac{\sum f_i x_i^2}{N} - \bar{x}^2 \right]^{\frac{1}{2}}$$

Table 3 A Typical Number Size Distribution

Class interval (μm)	6 to <7	7 to <8	8 to <9	9 to <10	10 to <11	11 to <12	12 to <13	13 to <14
Class midpoint (μm) (x_i)	6.5	7.5	8.5	9.5	10.5	11.5	12.5	13.5
Number of particles (f_i)	11	29	68	92	96	64	30	10

A-3 There may be occasions when a graphical representation of the size distribution is desirable. In this case, the use of a log-normal probability display (or normal probability display) is preferred to any other display form. In this type of display, one axis is ruled in a probability grid, the other in a logarithmic (or linear) fashion. The procedure for probability plot is given in **A-3.1**.

A-3.1 Rank the observations in increasing order. Let the observations, when arranged in increasing order, be denoted as $x_1, x_2, \dots, x_n$. Plot the x_i on the log-normal probability paper versus $(2i - 1) 50/n$. Here, it is assumed that marked axis on the probability paper refers to the percent of observations. The axis of the graph paper on which the x_i 's are plotted will be referred to as the observational scale, and the axis for $(2i - 1) 50/n$ as the cumulative scale. If a straight line appears to

fit the data, draw such a line on the graph paper. If the data is fitting to a log-normal distribution, then the points should cluster around this line although there will be some deviations because of random sampling fluctuations.

The advantage of this type of probability plot is that it enables the median sizes and standard deviation to be determined by inspection since the median is the intercept at 50 percent point of cumulative scale. The standard deviation is obtained by dividing the intercept between 5 percent and 95 percent values by 3.23. The values of median and standard deviation so determined will correspond approximately but not exactly with the calculated values. The graphical method shall, therefore, be used only to give an approximate result for rapid assessment of a powder. In all such cases, the calculated values are definitive.

ANNEX B

(Clause 5.2)

THE MICROSCOPE, IMAGE GENERATION AND GRATICULE DESIGN

B-1 CHARACTERISTICS OF THE MICROSCOPE

B-1.1 The microscope shall be of good quality and provided with coarse and fine focussing, focussing and centring sub-stage condenser, an adjustable sub-stage condenser diaphragm, and a mechanical stage giving to graduated movements at right angles to each other. The microscope shall be of the projection optical type (or scanning or transmission electron type for smaller sizes).

B-1.2 The illumination sources which may be integral with the microscope, shall be capable of filling uniformly the whole field of view of the lowest power objective used. It shall be provided with an adjustable diaphragm or other means of controlling the area of the field illuminated.

B-1.3 The objectives shall be of good quality, in sound condition and be inscribed with their focal length and numerical aperture (NA).

The objectives may be achromatic (or parachromatic), semi-apochromatic, fluorite or apochromatic. A number of different objectives will be required to provide the necessary magnification outlined. Care shall be taken to ensure that objectives have been corrected for the cover-glass thickness and tube length in use.

B-1.4 One or more eye-pieces are required and they shall be of good quality and in sound condition. They may be Huygenian (negative) or compensating (negative or positive) type. Compensating eye-pieces shall be employed with apochromatic, fluorite and the higher power achromatic objectives.

B-1.5 The sub-stage condenser shall be of good quality and in sound condition. The condenser shall be of adequate numerical aperture so that the illuminating rays pass through the slide at all angles up to the maximum which the objective is able to accept. A condenser for use with an objective of NA higher than 0.6 shall, in addition to being aplantic, be achromatic. It

may be necessary to make available more than one sub-stage condenser since it may not be possible to illuminate the whole field of view of low power objectives by means of a condenser having a numerical aperture adequate for high power objectives.

B-1.6 The stage micrometer used for magnification verification shall be divided into divisions of 10 μm . The glass slide carrying the micrometer shall be thin enough to permit proper adjustment of the sub-stage condenser. The stage micrometer shall be certified by an appropriate laboratory or regulatory body.

B-1.7 In operation, care shall be exercised to ensure proper alignment of all the components to the optical axis of the objective in use. There shall be no lateral movement of the particle images when the objective is racked up and down through the position of focus. The full field of view shall be uniformly filled with light over the area occupied by the graticule. It is equally important that the full numerical aperture of the microscope is uniformly illuminated. To fill the whole aperture, it is essential, firstly that the illumination cone angle of the sub-stage condenser shall be at least as large as that which the objective can accept, and secondly that the whole of the back lens of the condenser shall be uniformly illuminated. The intensity and the colour of the illumination shall be adjusted by means of neutral and coloured filters. The level of illumination should be correctly chosen, that is, high enough for adequate vision and low enough to prevent eye fatigue.

B-1.8 When adjusting to a selected magnification, it is important to verify that the magnification is uniform in different direction. The final screen magnification shall be verified before and after performing a sizing operation.

B-1.9 Mercury or halogen-arc lamps are rich in ultra-violet radiation, which can be harmful to human eyes. When employed, therefore, they shall be used with care and it is recommended that appropriate ultra-violet filters be used in the light path. The light source should also be properly enclosed to prevent the escape of unfiltered radiation.

B-2 MAGNIFICATION SELECTION

B-2.1 Because of the wide variety of instruments in the market, it is impossible to specify exactly the magnification to be used for all possible systems. The following provides guidelines for the choice of magnifications and the specified minimum requirements:

- a) The magnification shall not be too high. Otherwise, there will be a circle and particle image by eye can be done

without loss of resolution. Such loss in resolution may occur if the minimum detectable difference in circle and particle image sizes is too large a proportion of the total particle image size. (At best the eye cannot resolve to better than about 0.1 mm at 25 cm distance).

- b) The magnification shall not be too high. Otherwise, there will be an inadequate number of particles visible in the image at one time and the graticule sizes become inconveniently large.

- c) Numerical aperture of the objective lens shall be sufficiently large. Ideally, the final magnification should be between 500 and 1 000 times the value of the numerical aperture. This range is the so-called useful magnification range. Final values exceeding this limit appreciably will result in empty magnification while final magnifications below the limit do not make full use of the potentialities of the optics. Experience has shown that in the examination of diamond micron powders, it is better to err on the side to empty magnification but in no case may a numerical aperture less than the
$$\frac{\text{magnification}}{2\,000}$$
 be used, and as far as possible, it should lie in the range
$$\frac{\text{magnification}}{500} \text{ to } \frac{\text{magnification}}{100}.$$

- d) The diameters of the circles on the measurement graticule shall have a proper relation to the magnification. For sizes larger than M 2.5, the circles must define at least 8 equal size intervals in the main size range (from about 0.67 to about 1.33 $\times$, where $\times$ is the mean size) and may conveniently also define one or two equal size intervals immediately above and below the main size range.

There shall also be a circle for defining the oversize limit. This may be on the same or on a different graticule.

- e) The optical microscope is inadequate for checking sizes smaller than M 4.0. Ideally, scanning or transmission electron microscopes should be used for this range. However, these instruments are expensive and not commonly available in laboratories checking diamond micron powders. Because of the commercial importance of these sizes, it has been customary to check them with the optical microscope. This is permissible in the absence of better alternative methods but is subject to limitations imposed by physical optics and the wave length of visible light. Image quality below about 2 microns particle

size is very poor and for the finest sizes, the optical microscope may only give useful information about compliance with the oversize limit.

B-3 MICROSCOPE SYSTEMS

B-3.1 The following microscope systems may be used:

- a) Projection optical microscopes with continuously variable magnifications;
- b) Projection optical microscopes with fixed magnification steps;
- c) Scanning or transmission electron microscopes for sizes smaller than M 4.0; and
- d) Systems for the manual or semi-automatic analysis of photographs taken with (a), (b) or (c).

Microscope systems which rely on unidirectional measurement (for example Feret, diameter) may not be used. These include hair line eyepiece gratitudes and image splitting devices.

B-3.2 Projection Optical Microscope with Continuously Variable Magnification

The magnifications indicated in Table 2 shall be used. They shall be accurately set and checked with a stage micrometer before and after measurement.

Sizes M 4.0 to M 40 may then be measured with one graticule ruled with circles of inner diameters increasing in 1 mm steps from 3 to 17 mm, plus circles of 20 to 25 mm diameter for oversizes checking. However, it may be convenient to scan for oversize particles at lower magnification since larger number of particles may then be observed.

For sizes smaller than M 4.0, a graticule with finer divisions is necessary. One ruled with circles of diameters increasing in 0.5 mm steps from 1.0 to 11 mm is convenient. A circle of 16 mm diameter is needed for checking the harmful oversize limit for the size M 2.5 but is available on the graticule for the larger sizes, and again lower magnification may be used in scanning for harmful oversize. The use of the finer graticule is limited by the considerations set out in B-2.1 (e).

B-3.3 Projection Optical Microscopes with Fixed Magnification

The general considerations of the previous section apply. The actual magnifications used shall be accurately determined and checked before and after measurement of diamond powder by the use of a stage micrometer. They shall not be less than 80 percent of the values shown in Table 2 nor more than 160 percent of these values.

B-3.4 Electron Microscopes

These give the possibility of much improved resolution and increased accuracy of size measurement for the finer sizes. They may not be used for sizes M 4.0 and larger. They may be used for the size M 2.5 and smaller but proper attention shall be paid to technique. It is not the purpose of this standard to describe electron microscope techniques in detail. They are complicated, and reference should be made to standard text books for the details. It is emphasized, however, that there is substantial possibility of error in sample preparation methods which may concentrate certain size fractions selectively and also in magnification calibration. If electron microscope methods are used, they shall be performed by competent electron microscopists with full attention to these matters.

B-3.5 Use of Photographic Techniques

Rather than measuring the microscopic image directly, it may be convenient to record it photographically. The photographs may then be analysed by the use of a superposed transparent graticule. There are also instruments which project a light spot of a variable diameter on to a photograph. The spot is adjusted to match the particle size and the diameter may then be recorded automatically. These systems are permissible.

- a) The final magnification of the photograph at the time of measurement is directionally uniform and accurately known (some types of photographic processors) (for example, rotary dryers) cause unidirectional stretching of the print.
- b) The number of size intervals in the main range is at least 8 [see B-2.1 (d)].

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